

## Phosphorus Extractability of Soils Amended with Stockpiled and Composted Cattle Manure

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### ABSTRACT

Managing fertilizer applications to maintain soil P below environmentally unacceptable levels should consider the contribution of manure and synthetic fertilizer sources to soluble and extractable forms of P. Our objective was to evaluate soil and manure characteristics and application rates on P extractability in recently amended soils. Five soils of the U.S. southern High Plains were amended with beef cattle manures, composted beef manure, and inorganic fertilizers [ $\text{Ca}(\text{H}_2\text{PO}_4)_2$  or  $\text{KH}_2\text{PO}_4$ ] at five rates and incubated under controlled conditions. Mehlich 3-, Olsen ( $\text{NaHCO}_3$ )-, Texas A&M extractant (TAM)-, and water-extractable P were determined for the soils after selected incubation periods. Except for TAM and some water extractions, P extractability as a function of total P applied was linear ( $P < 0.001$ ) for a wide range of application rates. Mehlich-3,  $\text{NaHCO}_3$ , and water P extraction efficiencies of  $\text{KH}_2\text{PO}_4$ -amended soils averaged 22, 34, and 115% greater ( $P \leq 0.036$ ), respectively, than efficiencies of soils amended with manures except for the Texline (calcareous) loam and Pullman clay loam soils. Phosphorus extraction efficiencies decreased with time for  $\text{KH}_2\text{PO}_4$ -amended soils ( $P < 0.05$ ) but remained stable or increased for manure-amended soils during the 8-wk incubation period. Across all soils and manure sources, changes in water-extractable P per unit increase in Mehlich 3-,  $\text{NaHCO}_3$ -, and TAM-extractable P averaged 100, 85, and 125% greater, respectively, for inorganic as compared with manure-amended soils. These source-dependent relationships limit the use of agronomic soil extractants to make correct inferences about water-extractable P and dissolved P in runoff.

IN REGIONS DOMINATED by animal-based agriculture, confined animal operations generate large quantities of manure in localized areas. In most cases, the only economically viable option for disposal is land application. Phosphorus in animal manures usually exceeds levels required by crops when applied to meet the N demand. Because of the nutrient imbalance between N and P, repeated application of manures leads to soil P enrichment and increased environmental risks associated with elevated concentrations of dissolved P in runoff (Sharpley et al., 1999).

In response to excessive P in surface waters, many state agencies developed guidelines for manure applications aimed at reducing P in agricultural runoff. The strategies vary among states but typically employ a P-index approach or a soil P threshold to help delineate P loss vul-

nerability. A shared feature among all P management strategies is the use of an agronomic soil P test to assess and monitor changes in the soil's P status and establish management guidelines for reducing surface P loss (Sharpley et al., 2003). Agronomic soil P tests (e.g., Mehlich 3) are often correlated with environmental soil tests such as water-extractable P and in some cases with soluble P in runoff (Sims et al., 2002; Pote et al., 1996). However, these relationships may be altered when soils are amended with manure or fertilizer P. In recently amended soils, Sharpley et al. (2001) found that dissolved P in runoff was not related to Mehlich 3-extractable P but, rather, a function of the source and quantity of P applied.

The characteristics and maturity of stockpiled manure and compost (Henry and Harrison, 1996), competitive sorption between organic anions and phosphate (Kafkafi et al., 1988; Hue, 1991; Sharpley and Sisak, 1997), soil characteristics, kinetics of sorption and precipitation-dissolution reactions, and environmental conditions all may influence how much applied manure P is extractable during the first few months after an application. Inorganic Ca in manures added to soils also has a dominant role in regulating the readily available P pool (Siddique and Robinson, 2003). Moreover, the amount and forms of P extracted from soils vary widely among differing extractants (Thomas and Peaslee, 1973). The amount of manure applied that becomes available to plants during a growing season is probably influenced by similar factors and mechanisms. For manure of cattle fed on a high concentrate diet, plant availability of P within the first year after an application was estimated as 60% (Iowa State University, 2003), 75% (Moffitt et al., 1999), and 90% (Zhang et al., 2003). The wide range in estimated P availability from manures is probably indicative of some degree of uncertainty regarding the turnover of manure P in soils, the level of P in the diet (Ebeling et al., 2002), and regional differences in prevailing soils and environmental conditions. The objective of this study was to determine the influence of soil characteristics, manure characteristics (maturity), and application rates on the extractability of P in recently amended soils incubated with time.

### MATERIALS AND METHODS

Five soils of the southern High Plains were selected for use in the P extractability study (Table 1). The soils were collected from the Ap horizon (0–0.15 m) in fields with no recent (<5 yr) history of manure amendments. Bulk soil samples were air-dried, sieved (2 mm), and stored at  $-8^\circ\text{C}$  until required for incubations. Selected properties of soils are provided in Table 2.

Bulk samples of manure and commercially produced com-

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**Abbreviations:** CEC, cation exchange capacity; DRP, dissolved molybdate-reactive phosphorus;  $E_p$ , efficiency of phosphorus extraction; TAM, Texas A&M extractant.

**Table 1. Classification, location, and cropping history of soils used in the study.**

Soil series	USDA classification	Location in Texas	Elevation m	Cropping history
Pullman	fine, mixed, superactive, thermic Torreptic Paleustoll	Randall County; 35°11'28" N, 102°04'55" W	1169	dryland wheat–sorghum–fallow rotation, no fertilizers
Texline	fine-loamy, mixed, superactive, mesic Calcic Paleustoll	Hansford County; 36°10'5.7" N, 101°13'27.9" W	955	soybean followed by winter wheat planted in the fall, synthetic fertilizers as required
Acuff	fine-loamy, mixed, superactive, thermic Aridic Paleustoll	Lubbock County; 33°47'43.1" N, 102°00'50.1" W	1049	Conservation Reserve Program grassland for 10 yr, no fertilizers
Amarillo	fine-loamy, mixed, superactive, thermic Aridic Pleustalf	Howard County; 32°16'10" N, 101°29'26" W	770	dryland cotton, synthetic fertilizers
Harney	fine, smectitic, mesic Typic Argiustoll	Moore County; 35°51'44.2" N, 101°53'29.8" W	1107	irrigated corn, synthetic fertilizers as required

posted manure were obtained from a beef cattle feed yard located near Amarillo, TX. Fresh manure was collected by hand from feedyard pens and dried in a 40°C convective oven overnight. Manures from a freshly scraped pen (scraped manure, from pens scraped approximately once every 4 mo), a “new” (approximately 4 mo) stockpile (new stockpiled manure), and an “old” (>1 yr) stockpile (old stockpile manure) were also collected from the same feed yard. Composted manure (compost) was prepared with scraped manure in windrows that were periodically watered and mechanically aerated over a duration of 2 mo. Additional organic amendments were not used in the production of the composted manure. Scraped, stockpiled, and composted manures were allowed to air-dry to a water content sufficiently low enough to permit processing. Manures were ground by hand with a mortar and pestle to pass through a 0.5-mm sieve and stored at –8°C. Selected properties of manures used are provided in Table 3.

### Incubation I

A three-way factorial experiment was used to evaluate the influence of the temperature–moisture regime, fertilizer source, and P rate on P extractability in a Pullman soil. Eighty grams of soil (105°C oven-dry equivalent) were weighed into 100-mL plastic containers into which manures and inorganic fertilizer were added at rates of 0, 31.25, 62.5, 125, and 250 mg total P kg<sup>–1</sup> soil. Unamended treatments were replicated six times and all other treatments were replicated three times. Inorganic fertilizer consisted of a solution of monocalcium phosphate [Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub>], KNO<sub>3</sub>, and NH<sub>4</sub>NO<sub>3</sub> that supplied the nutrients N, P, and K at a ratio of 3.0:1.0:1.1. After addition of manure P sources (fresh manure, new stockpile manure, old stockpile manure, and compost) to containers, they were mixed with the soil using a glass rod. Soils were incubated under either (i) a constant 25°C temperature regime or (ii) a repeated wetted (1 wk at 25°C) and drying (1 wk at 40°C) conditions. Deionized water was replaced weekly for both incubation regimes to attain –33 kPa water content. Container lids were fitted loosely over samples incubating at 25°C to permit free air exchange. Lids were removed from sample containers incubating at 40°C. All incubations were initiated at the 25°C temperature regime and

terminated 9 wk later at the end of a 25°C regime. Incubated samples were then air-dried at 40°C in a convective oven, ground to pass through a 2-mm sieve, and stored at –8°C for later analyses.

### Incubation II

Five selected soils of the southern High Plains region were amended with freshly scraped manure, composted manure, and inorganic fertilizer at five rates in triplicate and incubated under repeated wetted–drying conditions. Fertilizers were added to 70 g (oven-dry equivalent) of soil in 100-mL plastic containers at rates of 0, 20, 40, 80, and 120 mg total P kg<sup>–1</sup> soil. The inorganic fertilizer consisted of a solution of monopotassium phosphate (KH<sub>2</sub>PO<sub>4</sub>) and NH<sub>4</sub>NO<sub>3</sub> that supplied N, P, and K at a ratio of 2.5:1.0:1.26. Unamended treatments were replicated six times and all other treatments were replicated three times. Amended soils were incubated under repeated wetted (1 wk at 25°C) and drying (1 wk at 40°C) conditions as described for Incubation I. Half the experimental units (soil containers) were incubated for only 2 d at 25°C and then transferred to the 40°C environmental chamber for 5 d (1-wk duration). The remaining containers were incubated for 8 wk beginning with the 25°C temperature and terminating with 1 wk in the 40°C drying regime (8-wk duration). Upon termination of the 1- and 8-wk incubations, samples were air-dried at 40°C in a convective oven, ground to pass through a 2-mm sieve, and stored at –8°C for later analyses.

### Soil and Manure Analyses

Phosphorus was extracted from incubated soils with deionized water (2 g of soil in 20 mL, shaken end over end for 30 min), Mehlich-3 extractant (Mehlich, 1984; 2 g of soil in 20 mL, shaken end over end for 5 min), 0.5 M NaHCO<sub>3</sub> at pH 8.5 (Olsen et al., 1954; 1 g of soil in 20 mL, shaken end over end for 30 min), and the Texas A&M extractant (TAM) consisting of 0.025 M EDTA, 1.0 M HCl, and 1.4 M NH<sub>4</sub>OAc (Hons et al., 1990; 1 g of soil in 20 mL, shaken end over end for 45 min). Water and 0.5 M NaHCO<sub>3</sub> soil suspensions were filtered through Whatman (Maidstone, UK) no. 42 filter

**Table 2. Selected physical and chemical properties of soils.**

Soil series	Particle size distribution				pH (1:1)	CaCO <sub>3</sub> equivalents	NaOAc CEC†	NH <sub>4</sub> OAc-extractable bases					Total C	Total N	Mehlich-3 P
	Sand	Silt	Fine clay	Clay				Ca	Mg	Na	K	Total			
	g kg <sup>-1</sup>					g kg <sup>-1</sup>		cmol <sub>c</sub> kg <sup>-1</sup>					g kg <sup>-1</sup>		mg kg <sup>-1</sup>
Acuff	564	212	124	224	7.0		13.6	10.1	2.2	0.1	1.3	13.7	7.3	0.74	12.2
Amarillo	812	76	57	112	8.2	1	7.4	11.7‡	0.6	0.1	0.5	12.9	2.2	0.23	15.9
Harney	521	265	111	214	7.3		15.0	9.9	4.5	0.3	1.7	16.4	11.0	1.13	134.4
Pullman	172	447	147	381	6.8		27.5	18.0	5.6	0.2	2.1	25.9	10.2	1.00	43.7
Texline§	277	486	58	237	8.2	31	20.5						17.5	1.34	54.0

† CEC, cation exchange capacity determined by saturation of exchange sites with Na<sup>+</sup> at pH 8.2.

‡ Free carbonates are probably responsible for the larger than expected value of “exchangeable” Ca reported for the Amarillo soil.

§ Extractable bases not reported for the Texline soil because of significant quantities of free carbonates.

**Table 3. Selected characteristics of manures used in the incubation experiments.<sup>†</sup>**

Manure	Total			N to P ratio	DRP <sup>‡</sup>	Total					
	C	N	P			Ca	Mg	K	Na	Fe	Mn
	g kg <sup>-1</sup>					g kg <sup>-1</sup>				mg kg <sup>-1</sup>	
Fresh	463	27.5	4.53	6.07	0.325	19.1	5.28	9.6	4.70	242	148
Scraped	219	19.9	5.14	3.87	0.431	25.7	3.82	26.3	6.21	223	272
New stockpile	188	25.3	8.40	3.01	0.361	29.5	5.59	31.4	7.34	257	359
Old stockpile	168	19.4	6.26	3.10	0.445	29.8	4.46	28.0	6.01	250	302
Compost	147	17.4	6.26	2.78	0.489	28.5	4.72	32.4	7.20	243	376

<sup>†</sup> All concentrations are on a dry-matter basis and represent an average of five (C, N, P, and DRP) and two (Ca, Mg, K, Na, Fe, and Mn) replicates.

<sup>‡</sup> Dissolved molybdate-reactive phosphorus from a 1:10 extraction with deionized water with a 30-min shaking time.

paper and Mehlich-3 and TAM suspensions were filtered through Whatman no. 2 filter paper.

Manures were digested with sulfuric acid and hydrogen peroxide for determination of total P in digests (Richards, 1993). Dissolved molybdate-reactive phosphorus (DRP) in manures was determined by shaking 2 g of manure in 20 mL of deionized water end over end for 30 min and filtering with a vacuum through a 0.45- $\mu$ m membrane filter. The DRP in extracts and digests was determined using a modified colorimetric molybdate-blue method of Murphy and Riley (1962) in conjunction with an autoanalyzer (USEPA, 1983). The Murphy and Riley (1962) method measures a portion of the acid-hydrolyzable organic P in addition to all orthophosphate P present in extracts (Haygarth and Sharpley, 2000). Total elemental C and N in manures and soils were determined by dry combustion and subsequent thermal conductivity analysis of evolved gasses using an Vario Max CN analyzer (Elementar, Hanau, Germany). Mineralized carbon was determined as total C in the manures added (Table 3) plus the initial total C in soils (Table 2) less the total C of incubated soils. Water contents of soils at  $-33$  kPa were determined using a pressure plate apparatus. Particle size distribution of soils was determined by wet-sieving and the pipette method (Gee and Bauder, 1986). Calcite and dolomite content of soils were determined using a Chittick apparatus (Dreimanis, 1962). Cation exchange capacity was determined by the Na-OAc (pH 8.2) procedure of Rhoades (1982) and pH was measured using a 1:1 soil to water ratio. Sorption isotherms were generated by fitting the Langmuir equation to 8-wk water-extractable P data and estimating previously sorbed P using a least-squares fit method (Reddy et al., 1998).

### Statistical Analyses

Statistical analysis was completed using the general linear models procedure (SAS Institute, 1999) to test for significant treatment effects. Orthogonal polynomial contrasts were used to test for linear, quadratic, and cubic trends between P application rate and soil test P. The efficiency of phosphorus extraction ( $E_p$ ) was calculated as the slope of the linear regression of extractable P as a function of applied P. Significant differences in linear trends between different fertilizer P sources, incubation regimes, incubation times, and soils were tested using mutually orthogonal linear regression contrasts of the interactions (Schabenberger and Pierce, 2002). Regression analyses were also used to study the relationships between P extracted by different soil tests.

## RESULTS AND DISCUSSION

Extractable P increased with increasing P application rate for all treatments in both incubation experiments

as exemplified by the Acuff soil at 8 wk (Fig. 1). The trend of extractable P in response to applied P was highly linear ( $P < 0.001$ ) for all treatments and extractants. Orthogonal polynomial contrasts also yielded significant ( $P < 0.05$ ) positive quadratic trends for all P sources with the TAM extractant and for  $\text{KH}_2\text{PO}_4$  with deionized water. This signifies that the proportion of total P extracted increases with increasing application rates. Hons et al. (1990) also obtained positive quadratic trends for the TAM extractant on noncalcareous soils. The Texline soil also exhibited a positive quadratic response ( $P < 0.05$ ) to applied  $\text{KH}_2\text{PO}_4$  with the  $\text{NaHCO}_3$  extractant (data not shown). The  $E_p$  values varied widely depending on the extractant, soil, and P source (Tables 4 and 5). Analysis of variance for Incubations I and II indicated highly significant main effects and two factor interactions for  $E_p$  among all extractants. Because of highly significant interactions and masked (crossed) effects, separate two-way analysis of variance and the pertinent regression contrasts were performed to evaluate the influence of P source, incubation regime, time, and soil on the linear response of extractable P.

### Incubation Regime

Paired  $t$  tests (not shown) demonstrated that a wetted-drying incubation regime significantly ( $P < 0.001$ ) increased Mehlich 3- and water-extractable P of unamended Pullman soil as compared with a constant temperature regime with minimal drying. A small but consistent increase in native extractable soil P following drying has also reported for EDTA,  $\text{NaHCO}_3$ , deionized water, and resin extractions (Haynes and Swift, 1985; Pote et al., 1999; Turner and Haygarth, 2001, 2003). While Mehlich 3- and water-extractable P of unamended Pullman soil was greater ( $< 8$  mg kg<sup>-1</sup>) under a wetted-drying regime as compared with the constant temperature regime, this trend was reversed for Pullman soils amended with stockpiled manure and resulted in greater P extractabilities under a constant temperature regime at the higher application rates. Consequently, efficiency of P extraction for both Mehlich 3- and water-extractable P was greater ( $P \leq 0.043$ ) for stockpiled manure-amended Pullman soil incubated under a constant temperature regime as compared with a wetted-drying regime. Decreased extractability of P under a wetted-drying regime may be a result of lower water contents that slowed the mineralization of the substrate.



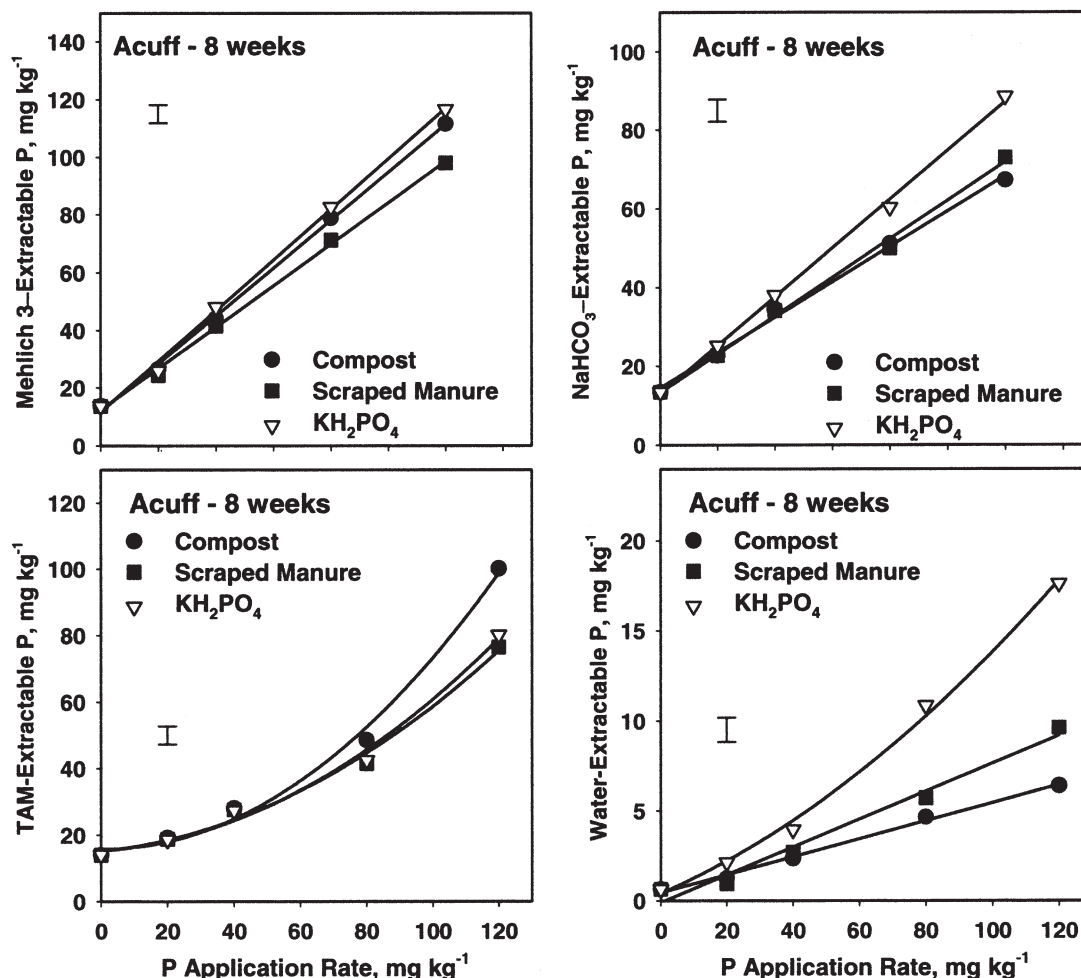


Fig. 1. Mean extractable P as a function of applied P for the Acuff soil at 8 wk (Incubation II). Error bars are 95% confidence limits of least-squares means for amended soils.

### Time

With few exceptions, differences between  $E_p$  at 1 and 8 wk were dominated by significant ( $P < 0.05$ ) decreases with time for KH<sub>2</sub>PO<sub>4</sub>-amended soils (Table 6). Decreases in Mehlich-3, Olsen, and water  $E_p$  with time (23, 39, and 64%, respectively) were greatest for the KH<sub>2</sub>PO<sub>4</sub>-amended calcareous Texline soil (Table 5). Lack of time dependence for TAM  $E_p$  in the KH<sub>2</sub>PO<sub>4</sub>-amended Texline soil (Table 6) probably results from the highly buffered acidic TAM extractant in conjunction with large solution to soil ratios and long shaking times sufficient to dissolve most of the recently precipitated calcium phosphates (Ca-P).

In contrast to KH<sub>2</sub>PO<sub>4</sub>-amended soils, significant changes in  $E_p$  with time for soils amended with composted or scraped manure were more infrequent and resulted in  $E_p$  increasing with time (Tables 5 and 6). On average, 37 and 48% of the organic C in added scraped manure was lost (mineralized) at the end of the 1- and 8-wk incubation periods, respectively (data not shown). Smaller fractions of organic C in added composted manure were mineralized at the end of the same incubation periods (17 and 28% after 1 and 8 wk, respectively). Because P mineralization rates are typically correlated

with C mineralization (Dao and Cavigelli, 2003), increased extractability with time of manure-amended soils probably resulted from mineralization of organic P in manures. Release of humic acids and organic anions by the decomposition process may also have (i) formed complexes with Ca (Ca-humate) thereby reducing solution Ca and increasing P solubility (Dalton et al., 1952; Moreno et al., 1960) and (ii) blocked or occupied sites of orthophosphate sorption on the anion exchange complex (Kafkafi et al., 1988).

Table 4. Summary of P extraction efficiencies for the Pullman soil under constant and wetted-drying regimes for Incubation I.

P source	Regime	Efficiency of P extraction†			
		Mehlich 3	NaHCO <sub>3</sub>	TAM	Water
Compost	constant	0.83	0.45	0.74	0.087
	wet-dry	0.74	0.49	0.73	0.082
Fresh manure	constant	0.48	0.47	0.41	0.069
	wet-dry	0.50	0.42	0.42	0.058
New stockpile manure	constant	0.72	0.37	0.69	0.095
	wet-dry	0.66	0.45	0.71	0.082
Old stockpile manure	constant	0.71	0.38	0.69	0.108
	wet-dry	0.67	0.41	0.74	0.100
Ca(H <sub>2</sub> PO <sub>4</sub> ) <sub>2</sub>	constant	0.65	0.37	0.41	0.119
	wet-dry	0.66	0.44	0.44	0.112

† Calculated as the slope of extractable P (mg kg<sup>-1</sup>) as a function of P application rate (mg kg<sup>-1</sup>). TAM, Texas A&M extractant.

**Table 5. Summary of P extraction efficiencies for the soils and P sources used for Incubation II.**

			Efficiency of P extraction†			
Soil	Time	Source	Mehlich 3	NaHCO <sub>3</sub>	TAM	Water
	wk					
Acuff	1	compost	0.87	0.49	0.64	0.051
		scraped	0.67	0.42	0.47	0.072
	8	KH <sub>2</sub> PO <sub>4</sub>	0.90	0.72	0.66	0.193
		compost	0.83	0.45	0.68	0.050
Amarillo	1	scraped	0.72	0.49	0.52	0.079
		KH <sub>2</sub> PO <sub>4</sub>	0.88	0.62	0.55	0.148
	8	compost	0.80	0.57	0.77	0.075
		scraped	0.65	0.48	0.64	0.108
Harney	1	KH <sub>2</sub> PO <sub>4</sub>	1.06	0.87	0.87	0.292
		compost	0.88	0.56	0.78	0.080
	8	scraped	0.73	0.48	0.60	0.112
		KH <sub>2</sub> PO <sub>4</sub>	1.06	0.68	0.88	0.220
Pullman	1	compost	0.83	0.48	1.05	0.113
		scraped	0.67	0.45	0.89	0.146
	8	KH <sub>2</sub> PO <sub>4</sub>	1.13	0.77	1.10	0.366
		compost	0.85	0.40	1.08	0.095
Texline	1	scraped	0.85	0.46	0.99	0.139
		KH <sub>2</sub> PO <sub>4</sub>	1.04	0.60	0.94	0.307
	8	compost	0.78	0.50	0.71	0.070
		scraped	0.64	0.45	0.57	0.076
Means	1	KH <sub>2</sub> PO <sub>4</sub>	0.93	0.64	0.59	0.177
		compost	0.88	0.50	0.68	0.087
	8	scraped	0.68	0.49	0.54	0.086
		KH <sub>2</sub> PO <sub>4</sub>	0.79	0.56	0.50	0.127
Means	1	compost	0.81	0.45	0.96	0.041
		scraped	0.63	0.47	0.73	0.080
	8	KH <sub>2</sub> PO <sub>4</sub>	0.85	0.55	1.13	0.148
		compost	0.83	0.50	1.01	0.057
Means	1	scraped	0.72	0.47	0.90	0.086
		KH <sub>2</sub> PO <sub>4</sub>	0.65	0.33	1.17	0.053
	8	compost	0.84	0.49	0.84	0.072
		scraped	0.70	0.47	0.69	0.098
Means	1	KH <sub>2</sub> PO <sub>4</sub>	0.93	0.63	0.84	0.203

† Calculated as the slope of extractable P (mg kg<sup>-1</sup>) as a function of P application rate (mg kg<sup>-1</sup>). TAM, Texas A&M extractant.

## Soil

Soil type explained 56 to 62% of the variability of extractable Mehlich-3, TAM, and water P under Incubation II (not shown). Variability of NaHCO<sub>3</sub>-extractable P due to soil type was less sensitive and accounted for only 31% of the total sum of squares. The effect of differences in initial extractable soil P among soils on the analysis of variance can be removed by subtracting mean P extractability of unamended samples from amended samples to yield *net extractable P* (Griffin et al., 2003). Repeating the analysis of variance using net extractable P resulted in percentages of soil sum of squares relative to the total sum of squares of approximately 2% for the Mehlich-3 and NaHCO<sub>3</sub> extractants and 11% for the

**Table 6. Linear regression contrasts testing for differences between time × rate interactions for Incubation II.†**

Soil	Source	P > F			
		Mehlich 3	NaHCO <sub>3</sub>	TAM‡	Water
Acuff	compost	0.329	0.201	0.085	0.771
	scraped manure	0.016	0.001	0.107	0.099
	KH <sub>2</sub> PO <sub>4</sub>	0.477	<0.001	<0.001	<0.001
Pullman	compost	0.045	0.941	0.379	0.003
	scraped manure	0.364	0.245	0.402	0.233
	KH <sub>2</sub> PO <sub>4</sub>	0.041	0.003	0.002	<0.001
Texline	compost	0.769	0.252	0.460	0.002
	scraped manure	0.052	0.676	<0.001	0.299
	KH <sub>2</sub> PO <sub>4</sub>	<0.001	<0.001	0.340	<0.001

† H<sub>0</sub>: Efficiency of phosphorus extraction (E<sub>P</sub>) at 1 wk = E<sub>P</sub> at 8 wk.

‡ Texas A&M extractant.

**Table 7. Selected linear regression contrasts for soil × rate interactions of Incubation II.**

		$P > F$			
Time	Source	Mehlich 3	NaHCO <sub>3</sub>	TAM†	Water
wk					
$H_0$ : calcareous $E_p$ = noncalcareous $E_{p\ddagger}$					
1	compost	0.682	0.061	<0.001	<0.001
	scraped manure	0.507	0.563	0.042	0.023
	KH <sub>2</sub> PO <sub>4</sub>	0.007	<0.001	<0.001	<0.001
8	compost	0.337	0.432	<0.001	<0.001
	scraped manure	0.541	0.791	<0.001	0.005
	KH <sub>2</sub> PO <sub>4</sub>	<0.001	<0.001	<0.001	<0.001
$H_0$ : $E_p$ of low CEC soils = $E_p$ of high CEC soils‡					
1	compost	0.472	0.196	<0.001	<0.001
	scraped manure	0.948	0.929	<0.001	0.003
	KH <sub>2</sub> PO <sub>4</sub>	0.425	<0.001	0.026	0.008
8	compost	0.637	0.024	<0.001	<0.001
	scraped manure	0.329	0.940	<0.001	<0.001
	KH <sub>2</sub> PO <sub>4</sub>	0.154	0.027	0.667	<0.001

† Texas A&M extractant.

‡ Soil classes are as follows: calcareous = Texline; noncalcareous = Acuff, Amarillo, Harney, Pullman; low cation exchange capacity (CEC) (<15 cmol<sub>c</sub> kg<sup>-1</sup>) = Amarillo, Acuff; high CEC (>15 cmol<sub>c</sub> kg<sup>-1</sup>) = Harney, Pullman.

TAM and water extractants. Thus, the initial P extractability of unamended soils accounted for a large proportion of the variability among soils. Moreover, the TAM and water P extractability were most sensitive to differences among soils.

Differences in E<sub>P</sub> among soil types were significant principally for the TAM and water extractions (Table 7). Generally, differences in response to added P were less evident for the Mehlich-3 and NaHCO<sub>3</sub> extractants except for KH<sub>2</sub>PO<sub>4</sub>-amended soils (Table 7). The KH<sub>2</sub>PO<sub>4</sub>-amended Texline, a calcareous soil, had significantly lower E<sub>P</sub> for the Mehlich-3, NaHCO<sub>3</sub>, and water extractants as compared with the four noncalcareous soils (Tables 5 and 7). In contrast, the TAM extractant yielded recovery rates of 100% of added P in the KH<sub>2</sub>PO<sub>4</sub>-amended Texline soil and greater than the E<sub>P</sub> of the four noncalcareous soils (Table 5).

Water-extractable P results for soils amended with synthetic fertilizer can be presented as sorption isotherms (Fig. 2). Note that equilibration time after P additions is 8 wk rather than the standard 24 h. For the four noncalcareous soils amended with KH<sub>2</sub>PO<sub>4</sub>, P sorption increased with increasing cation exchange capacity (CEC; Fig. 2). The calcareous Texline soil appeared to approximate a Langmuir isotherm until an equilibrium concentration of 0.028 mM L<sup>-1</sup> was attained, after which there was a change in slope (Fig. 2). Griffin and Jurinak (1973, 1974) demonstrated that a P concentration of 0.03 mM L<sup>-1</sup> was sufficient to cause orthophosphate sorption on calcite surfaces that was followed by heterogeneous precipitation of Ca-P and transformation to more stable, crystalline Ca-P phases. This mechanism is probably responsible for constraining solution P concentrations in the Texline soil.

## Phosphorus Source

Source by rate interactions (not shown) were highly significant (P < 0.001) except for the NaHCO<sub>3</sub> extractant of Incubation I indicating that, for most scenarios, E<sub>P</sub> differed among P sources. The P extraction efficiencies obtained with the four extractants ranged from 18 to

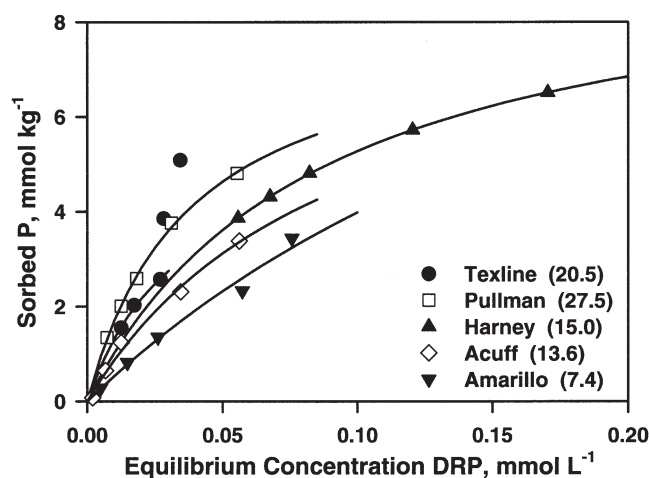


Fig. 2. Apparent sorption isotherms of orthophosphate P in  $\text{KH}_2\text{PO}_4$ -amended soils after 8 wk. Each point represents the mean of three measurements. Lines represent the nonlinear fits to the Langmuir equation. Values in parentheses are  $\text{NaOAc}$  cation exchange capacity (CEC;  $\text{cmol}_c \text{kg}^{-1}$ ) for each respective soil series. DRP, dissolved molybdate-reactive phosphorus.

52% greater for  $\text{KH}_2\text{PO}_4$ -amended Pullman soil (Incubation II at 8 wk) as compared with the  $\text{Ca}(\text{H}_2\text{PO}_4)_2$ -amended Pullman soil (Incubation I at 9 wk under a wetted-drying regime for P rates of 0 to 125  $\text{mg kg}^{-1}$ ). Depression of P extractability in  $\text{Ca}(\text{H}_2\text{PO}_4)_2$ -amended Pullman soil may have been caused by the precipitation of Ca-P as dicalcium phosphate dihydrate and subsequent conversion to insoluble secondary Ca phosphates (Bell and Black, 1970).

Differences in  $E_p$  among organic sources for the agronomic extractants Mehlich 3 and TAM were generally characterized as compost  $E_p \geq$  stockpiled or scraped manure  $E_p >$  fresh manure  $E_p$  (Tables 4, 5, and 8). Incompletely mineralized organic P in scraped, stockpiled, and especially fresh manures may be partly re-

sponsible for significantly lower  $E_p$  as compared with composted manure. Contrasts testing for differences in  $\text{NaHCO}_3$   $E_p$  among organic sources were generally non-significant. Despite lower agronomic P extractabilities, scraped manure-amended soils had greater water  $E_p$  as compared with compost-amended soils (Tables 5 and 8). Adler and Sikora (2003) similarly found that soil amended with immature (14 d) poultry compost had greater water-extractable P than soil amended with compost of greater maturity. These results may be related to the release of greater quantities of organic anions during the early decomposition phases of manure (Singh and Amberger, 1998) and consequently greater competition with orthophosphate for sorption sites.

Contrasts testing for differences between organic and inorganic P sources at 8 wk were significant except in a few cases for the Pullman soil (Table 8). Mehlich-3,  $\text{NaHCO}_3$ , and water P extraction efficiencies averaged 22, 34, and 115% greater ( $P \leq 0.036$ ), respectively, for  $\text{KH}_2\text{PO}_4$ -amended as compared with manure-amended Acuff, Amarillo, and Harney soils (Tables 5 and 8). In contrast, at 8 wk the calcareous Texline soil had a significantly greater ( $P \leq 0.002$ ) Mehlich-3,  $\text{NaHCO}_3$ , and water  $E_p$  when amended with scraped and composted manures as compared with  $\text{KH}_2\text{PO}_4$  amendments (Tables 5 and 8). Robbins et al. (2000) also observed greater 0.01 M  $\text{CaCl}_2$ -extractable P for a calcareous soil amended with dairy manure as compared with mono-calcium phosphate. Agronomic (Mehlich 3,  $\text{NaHCO}_3$ , and TAM)  $E_p$  values of the  $\text{Ca}(\text{H}_2\text{PO}_4)_2$  and  $\text{KH}_2\text{PO}_4$ -amended Pullman soil were typically lower than or equivalent to the corresponding  $E_p$  of the Pullman soil amended with composted, scraped, and stockpiled manure at 8 or 9 wk (Tables 4 and 5). Nevertheless, these inorganic amendments resulted in significantly ( $P < 0.001$ ) greater water P extractability for the Pullman as compared with manure amendments (Tables 4, 5, and 8).

An outcome of interactions occurring in these soils

Table 8. Level of significance for linear regression contrasts testing for differences between source  $\times$  rate interactions.

		<i>P &gt; F</i>			
Contrast <i>H</i> <sub>0</sub> †	Regime or soil	Mehlich 3	NaHCO <sub>3</sub>	TAM‡	Water
<u>Incubation I (Pullman soil)</u>					
Organic = inorganic§	constant	<0.001	0.110	<0.001	<0.001
	wet–dry	0.098	0.876	<0.001	<0.001
Aged = fresh§	constant	<0.001	0.047	<0.001	<0.001
	wet–dry	<0.001	0.110	<0.001	<0.001
Composted = stockpiled§	constant	<0.001	0.036	0.033	<0.001
	wet–dry	<0.001	0.014	0.636	0.040
Old stockpile = new stockpile	constant	0.606	0.844	0.988	0.009
	wet–dry	0.402	0.182	0.209	<0.001
<u>Incubation II (8 wk)</u>					
Compost = scraped	Acuff	<0.001	0.081	<0.001	<0.001
	Amarillo	<0.001	0.116	0.001	<0.001
	Harney	0.906	0.050	0.136	0.002
	Pullman	0.002	0.875	<0.001	0.830
	Texline	0.014	0.492	0.024	<0.001
Organic = inorganic¶	Acuff	<0.001	<0.001	0.001	<0.001
	Amarillo	<0.001	0.002	<0.001	<0.001
	Harney	0.036	<0.001	0.031	<0.001
	Pullman	0.802	0.007	<0.001	<0.001
	Texline	0.002	<0.001	<0.001	<0.001

† Efficiency of P extraction for the two groups of fertilizer sources is equivalent.

‡ Texas A&M extractant.

§ Groups of fertilizer sources for Incubation I are as follows: organic = all manures; inorganic =  $\text{Ca}(\text{H}_2\text{PO}_4)_2$ ; aged = compost, new stockpile manure, old stockpile manure; fresh = fresh manure; stockpiled manure = new stockpile manure, old stockpile manure.

¶ Groups of fertilizer sources for Incubation II are as follows: organic = composted and scraped manures; inorganic =  $\text{KH}_2\text{PO}_4$ .

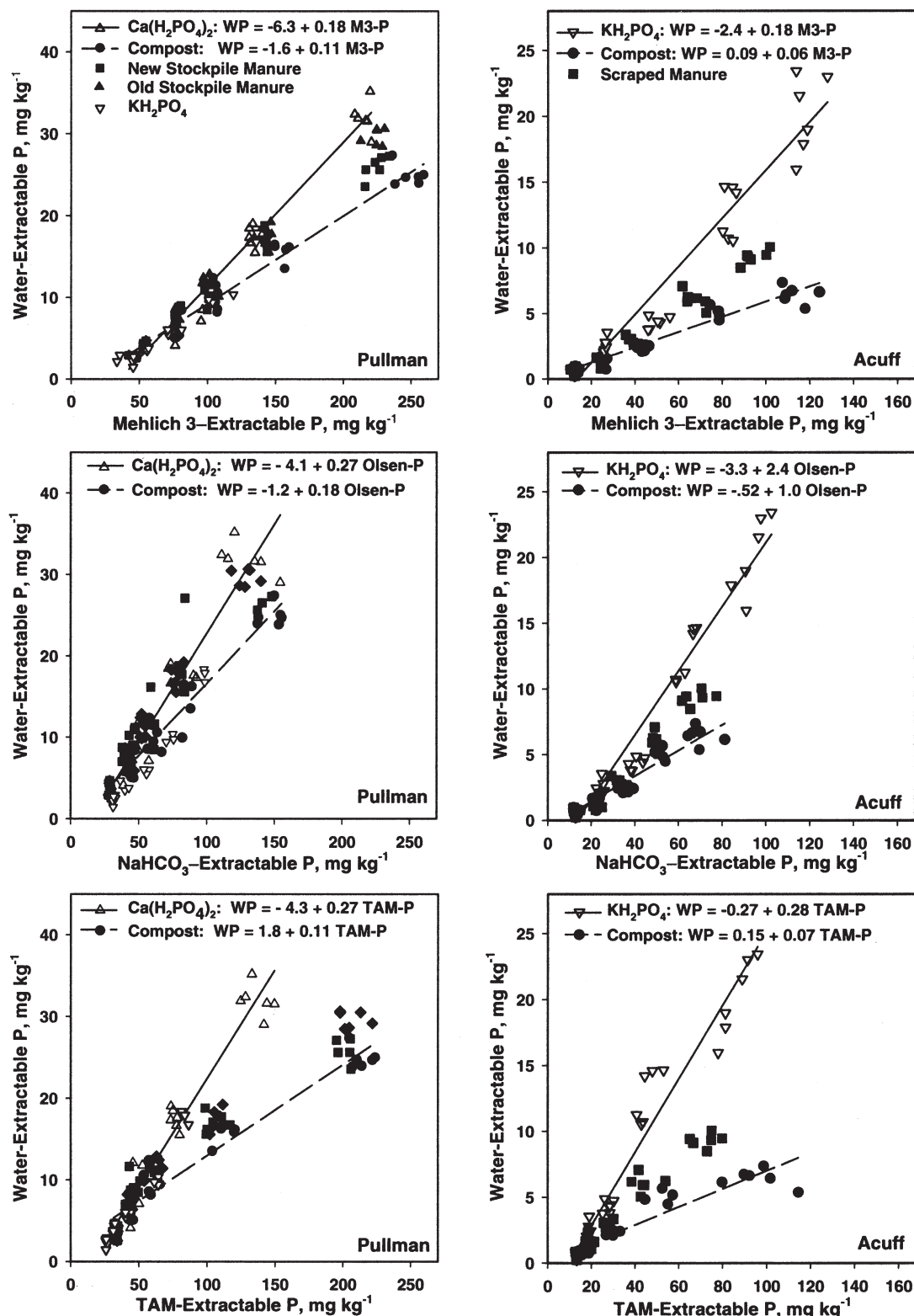


Fig. 3. Trends in Mehlich 3-,  $\text{NaHCO}_3$ -, and Texas A&M extractant (TAM)-extractable phosphorus (M3-P, Olsen-P, TAM-P) with water-extractable phosphorus (WP) for different fertilizer sources in the Pullman (Incubation I) and Acuff (Incubation II) soils. Linear regression equations are shown for  $\text{Ca}(\text{H}_2\text{PO}_4)_2$ ,  $\text{KH}_2\text{PO}_4$ , and composted manure P sources.

on addition of P was that the slopes of the relationship between water-extractable P and agronomic soil test P ( $\Delta\text{WEP}/\Delta\text{STP}$ ) were strongly dependent on the fertilizer P source. As compared with compost-amended soils,

$\text{Ca}(\text{H}_2\text{PO}_4)_2$ -amended soils exhibited 64, 51, and 140% greater increase in water-extractable P per unit increase in Mehlich 3-,  $\text{NaHCO}_3$ -, and TAM-extractable P, respectively, for the Pullman soil (Fig. 3). Similarly, changes



in water-extractable P per unit increase in Mehlich 3-,  $\text{NaHCO}_3$ -, and TAM-extractable P averaged 210, 140, and 310% greater, respectively, for  $\text{KH}_2\text{PO}_4$  as compared with compost-amended Acuff soil (Fig. 3). A greater  $\Delta\text{WEP}/\Delta\text{STP}$  for  $\text{KH}_2\text{PO}_4$  compared with manure-amended soils (>18%) was apparent for all soils and manures except for the TAM-extractable P of the scraped manure-amended Texline soil. Across all soils and manure sources, changes in water-extractable P per unit increase in Mehlich 3-,  $\text{NaHCO}_3$ -, and TAM-extractable P averaged 100, 85, and 125% greater for inorganic as compared with manure-amended soils, respectively.

A source-dependent correlation between agronomic and water-extractable P in this study may relate to the water extraction method and source-dependent P transformations throughout the incubation period. For these short-duration water extractions, soluble forms of Ca-P such as dicalcium phosphate dihydrate are probably not contributing much to solution P because of relatively slow dissolution rates of these minerals (Zhang et al., 1992; Jaynes et al., 2003). Consequently, surface-adsorbed phosphate may be contributing a greater proportion to orthophosphates in water extract solutions rather than Ca-P. Organic anions may compete with orthophosphates for surface reactions (Kafkafi et al., 1988) thereby decreasing P sorption in manure-amended soils. A greater proportion of orthophosphate saturation of surface sites on inorganic P-amended soils combined with fast desorption rates during the extraction period (Toor and Bahl, 1999) may explain why, for a given agronomic P extractability, water-extractable P was greatest for soils amended with  $\text{Ca}(\text{H}_2\text{PO}_4)_2$  and  $\text{KH}_2\text{PO}_4$  as compared with manure-amended soils.

## CONCLUSIONS

The increase in extractable P in soils amended with manures and synthetic fertilizers varied considerably with P source, incubation time, extractant, and soil characteristics. Except for the TAM extractions and some water extractions, P extractability as a function of total P applied was linear over a wide range of application rates. Lower P extraction efficiencies of the manure and  $\text{Ca}(\text{H}_2\text{PO}_4)_2$ -amended Pullman soil as compared with the  $\text{KH}_2\text{PO}_4$ -amended Pullman suggests that Ca in manures and fertilizers depressed P solubility and extractability. A larger proportion of applied P was extracted from composted manure as compared with stockpiled and fresh manures with the acidic, agronomic extractants (Mehlich 3 and TAM). Incompletely mineralized organic P in scraped, stockpiled, and especially fresh manures may be partly responsible for these lower efficiencies.

Changes in water-extractable P per unit increase in Mehlich 3-,  $\text{NaHCO}_3$ -, and TAM-extractable P were considerably greater for soils amended with synthetic fertilizers as compared with composted and scraped manure-amended soils. Water-extractable P is important because it reflects the degree of immediately bioavailable P and is closely related to dissolved P in overland flow (Pote et al., 1999). Although agronomic tests can also

be closely correlated with forms of soil P susceptible to runoff losses (Pote et al., 1996; Sims et al., 2002), in this study organic and inorganic P sources probably contributed to different soil P pools, which led to source-dependent relationships between agronomic soil test and water-extractable P. Consequently, use of agronomic soil tests such as Mehlich 3 to assess vulnerability to P loss over the short term may underestimate risks for soils amended with synthetic fertilizers and overestimate risks for soils amended with animal manures. As a result, these P source-dependent relationships limit the use of agronomic soil extractants to make correct inferences about water-extractable P and dissolved P in runoff in recently amended soils. These difficulties could be overcome by using both an agronomic and environmental soil test in a P indexing system so that both the degree of P saturation as well as readily desorbable and soluble P in soils are considered. An environmental soil test, such as water-extractable P, would only need to be considered if the agronomic soil test value is exceedingly high.

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